Taking QXAS data

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I. How it works and why use it

QXAS ("Quick XAS") data is collected by scanning the monochromator in a continuous motion and taking the fluorescence data on the fly, as opposed to the step-and-repeat mode of 'regular' XAS. This mode allows one to acquire scans in as little as 10 seconds. It is therefore possible to follow reasonably quick reactions. Also, if a sample damages easily under the beam, it is possible to limit the amount of time for which the beam dwells on any spot. In contrast, regular EXAFS can take up to 45 minutes for a single scan, which means that a sample could be damaged before the end of the first scan. A further advantage of QXAS is that spots on the sample seem to move at random on timescales of seconds. This effect can lead to a sample yielding hundreds of thousands of counts per second, but providing data that looks more appropriate to count rates of only a few kHz. QXAS essentially shifts the noise frequency spectrum and lets you get better results on jittery samples such as small particles on kapton tape. Similarly, a "drifty" sample may be better recorded in QXAS using a small number of sweeps with an auto-align step in between. This version of QXAS is not as fast as what's available at purpose-built QXAS lines (milliseconds) but is fast enough for many purposes.

Since a single scan rarely gives good enough signal to noise to be useful on its own, the QXAS program repeats the scan a number of times. The raw data are shown in a 2D intensity graph with each scan occupying a horizontal line, position along which representing energy. Thus, the QXAS dataset looks a little like an XRF map in which each line is a scan. Thus, we refer to scans within a single set as "lines".

The data are acquired using the same mapping mode as for XRF, except that instead of the X-stage clocking off gate pulses, the monochromator does it. I_0 is acquired using the "XBIC" mechanism wherein V/F pulses are converted into something like what comes out of a detector preamp and put into the XIA detector electronics. Thus, I_0 is recorded synchronously with fluorescence. Since I_0 is recorded in a different way than in either the normal EXAFS or mapping programs, it takes a different offset. Further, there is a frequency divider between the V/F and the converter box which is used to keep the

count rate in the I_0 channel in an acceptable range. This divider can be set to ÷1,÷10 or ÷10 . A note on the front panel of the QXAS program tells you which one to use.

Why not use QXAS all the time? For one, we can't yet do transmission because MAM hasn't yet built a second converter box. For another, the monochromator will only scan at a constant angular speed, so it is not possible to dwell longer at the high end of a scan. We will see that it is possible to vary the dwell time per point within a scan, but that means changing the energy resolution. Thus, if the step is set to 5eV in an energy region, the counts occurring within a 5eV band of energies will be accumulated to form the data point.

In order to save verbiage, we refer frequently to the regular EXAFS program and assume familiarity with its functions and controls. In what follows, that program will be referred to as SXAS ("slow" XAS).

II. Setting up

To set up for data acquisition, you need to establish the following:

1. Scaler map. This is like the one in SXAS in that it tells the program what to count, but different in detail.

2. Scan definition. Again, like SXAS but with QXAS-specific detail. Part of the scan definition is the choice of points on the sample.

3. I_0 divider setting and cabling. These items have no analog in SXAS.

These points will now be described in detail.

Like SXAS, the QXAS program has a scaler map and a scan definition. If you don't know what either are, please read up on them in the manual for SAXS. To generate an appropriate scaler map, use the Scaler map generator program. This lets you enter the ROI for the fluorescence channels. Be sure to select the QXAS separate detector (I0=8th channel) option. You only need to enter the ROI values; the I_0 ROI values are pre-set. The resulting scaler map is automatically written to the correct directory. You can use the same program to write EXAFS scaler maps as well.

The QXAS program has Operations and Plots pages just like the SXAS program. On the Operations page, you will find a note about I_0 counts and the divider box. Turn the knob on that box CCW for divide-by-1. The divider is in a NIM bin slot

and is obviously home-built. The input cable from that box should be plugged into the V/F converter (black module with lots of LEMO connectors) in the position marked QXAS. With the divider set as it usually is, a good range for I_0 is 0.5-2V. Go to the beamline-control computer, pull up the Amplifiers->Set up parameters window (the title bar for this window reads "Setup Current Amp Parameters.vi") and select High Bandwidth in the Gain Mode (top-left control). This minimizes the time lag between changes in I0 due to, for instance, glitches and the response of the current amp.

Once the scaler map and I_0 are set up, take offsets in the usual way. The button for this is on the Operations page.

The next thing is to set up a scan definition. There are buttons for loading, saving and editing a scan-def. The Scan Editor, which is accessed via a button on the Operations page, has several pages. One of these is for defining the energy regions:

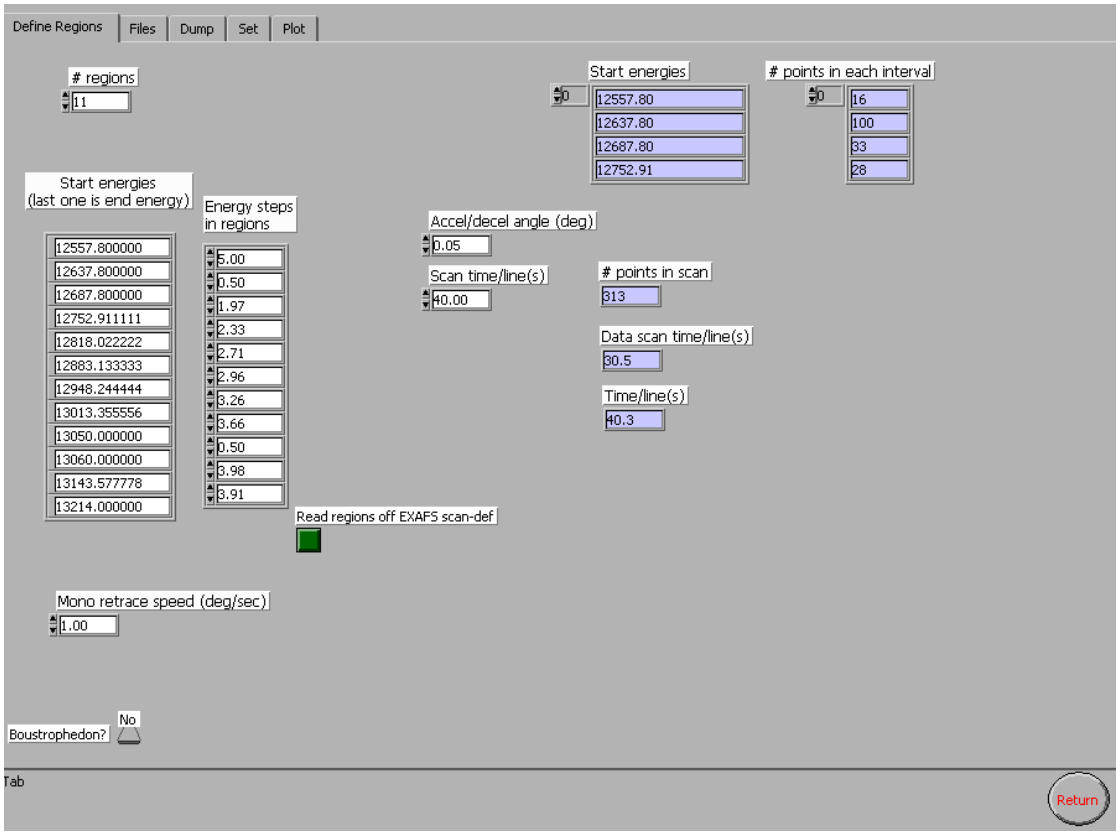

Figure 1. The Define Regions tab of the scan editor.

This page is much like its counterpart in SXAS but with some differences. One of these is that since the scan speed is constant, you can't define a separate time per point for each region. Instead, you tell it the scan time you want per line and it works out the requisite scan speed. Now, note the blue indicator for Data scan time/line (s). This number is less than the specified scan time because the scan starts below the nominal start energy and continues above the nominal end energy, in order to allow for acceleration and deceleration. The angle through which the mono moves during these over-scan periods is determined by the Accel/decel angle (deg) control, which you can leave at the default value of 0.05. Thus, we see that in this example, we lose about 10 seconds in these over-scan segments during which no data are taken.

As mentioned above, data collection is continuous between energy points. Thus, the last point represents data taken over a 3.91eV-wide band centered at 13214eV. By giving the last regions a bigger step than the first, we allow some signal averaging at the expense of energy resolution. How serious is the blur of the EXAFS pattern due to this averaging? Since $k(\text{Å}^{-1}) = 0.5132\sqrt{E(\text{eV}) - E_0(\text{eV})}$, we find $dk / dE\ (\text{Å}^{-1}) = 0.13 / k$. Now, suppose we want to have faithful recording of distances out to some $R_{\max}$, and assume that this means a phase shift of no more than $\pi / 2$ over the range *dk*. Doing the substitutions yields $dE(eV) \le 11.9k(\text{Å}^{-1}) / R_{\max}(\text{Å})$. For $k = 12.5\text{Å}^{-1}$ (500eV above the edge) and $R_{\max} = 10\text{Å}$, we find $dE \le 15\text{eV}$. Thus, the 4eV step used here is conservative and makes sense mostly in terms of not having large patches of data ruined by one-point glitches. The Read regions off EXAFS scan-def button lets you read from an existing SXAS scan-def, which makes for a good starting point.

The Boustrophedon? switch enables a mode in which the scan goes low to high emergy, then high to low, etc., saving retrace time. We don't tend to use it because there is an energy offset between the directions which isn't always consistent within the scan. There are controls in both the QXAS data-acquisition program and the data editor for compensation of constant offsets.

The Retrace speed is how fast, in degrees/second, the mono goes from the end of the scan line back to the beginning. This seems to be limited to 1^{o}/second by the maximum speed of the motor.

There are also File, Plot and Dump tabs, all of which are pretty similar to those in the ‘regular’ program.

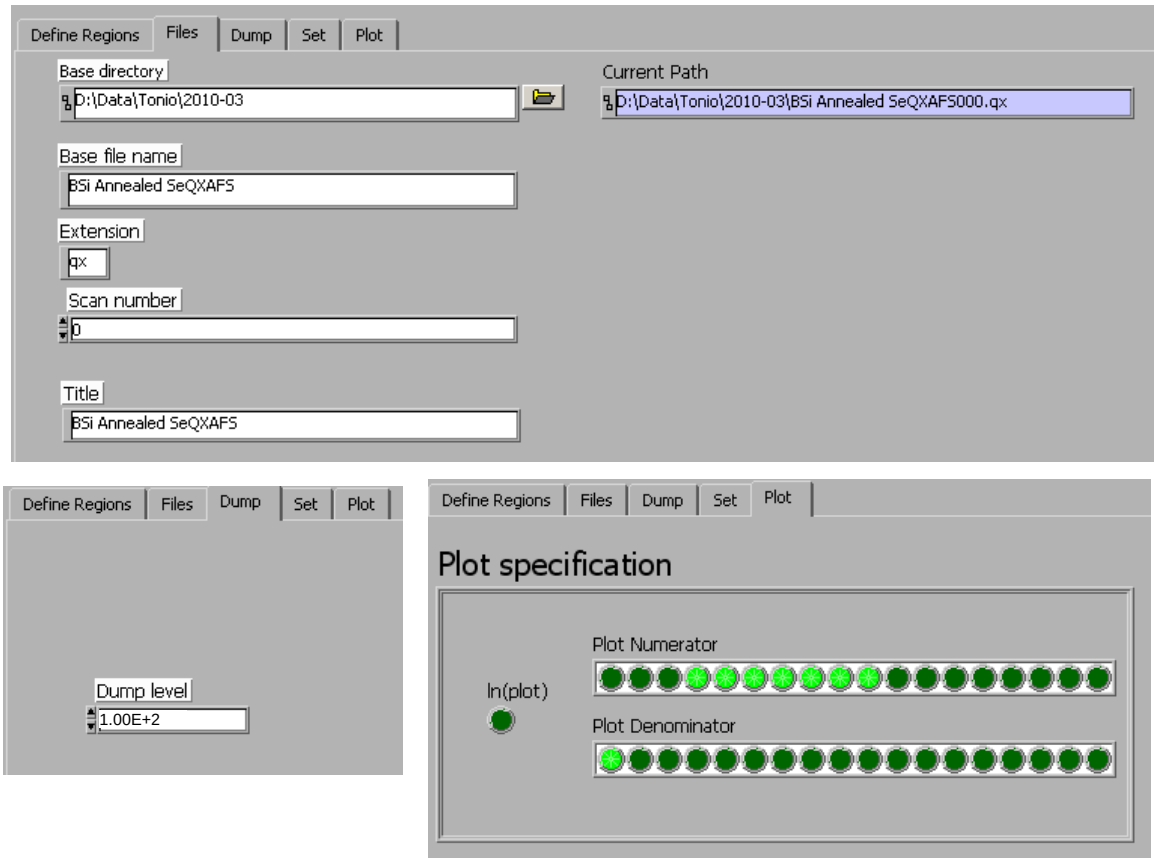

Figure 2. The Files, Dump *and* Plot *tabs of the scan editor.*

The Files tab works exactly as expected, except that the default extension is `.qx` instead of `.dat`. The Dump level is an absolute number of counts/second above the offset level, rather than a factor as in the other program. The Plot tab shows a different plotspec than the default in normal XAS. The channels, as defined in the scaler maps, are:

0 I_0
1 Fluorescence channel, all 7
2 Total counts, all 7
3-9 Fluorescence, detectors 0-6
10-16 Total counts, detectors 0-6
17 Total count, 8^{th} (I_0) channel

The final tab is the Set tab, which again works almost but not quite like its counterpart. The example shown below includes autoalign, which works exactly as it does in SXAS.

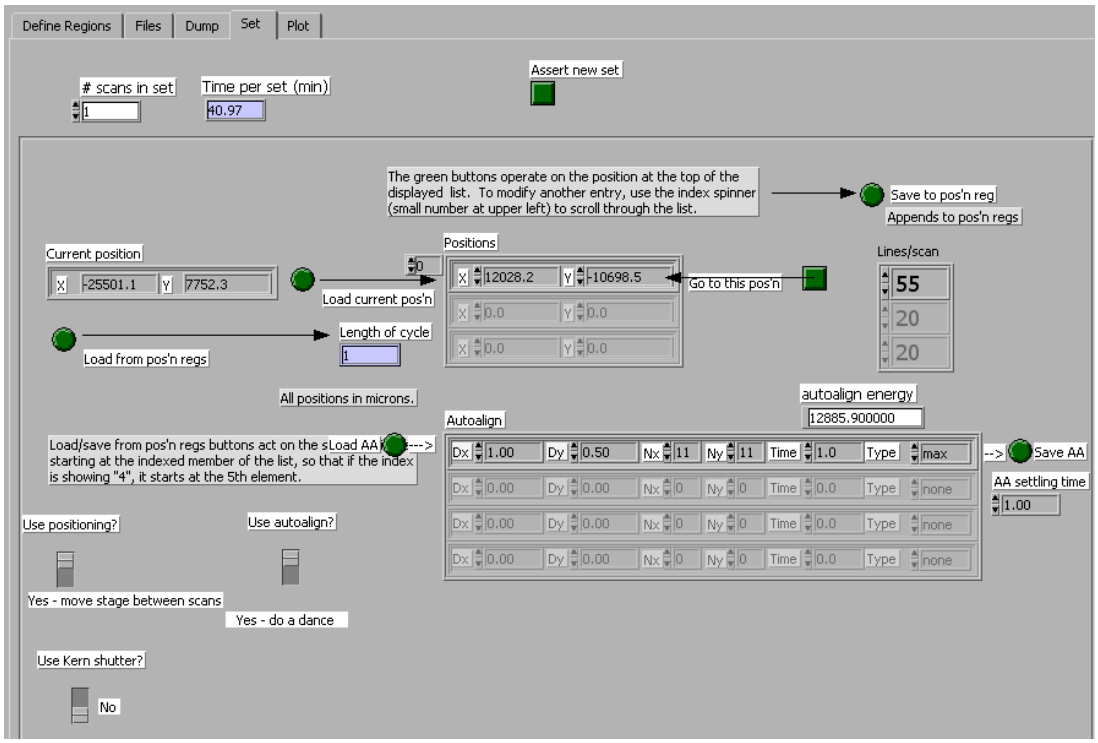

Figure 3. The Set *tab in the scan editor, showing Autoalign.*

The Lines/scan control sets how many energy sweeps it does at the given point. Thus, in the example, the `.qx` file will contain 55 lines's-worth of data for the one specified point. There is no Mono Freeze switch because the mono translate motion is always frozen in QXAS. The Use Kern shutter? switch enables the generation of a TTL pulse on one of the DIO channels which is intended to run a shutter which opens during data acquisition only. This feature was put in on request of Jan Kern.

As in SXAS, it is important to make sure that you specify the # of scans in set correctly to ensure that all desired points get recorded. There is no Repeat count option because increasing the Lines/scan takes care of that.

III. Running and monitoring data acquisition

The Plots page of the program looks like this during data-taking (data taken at Fe edge, not as specified in example scan-def above):

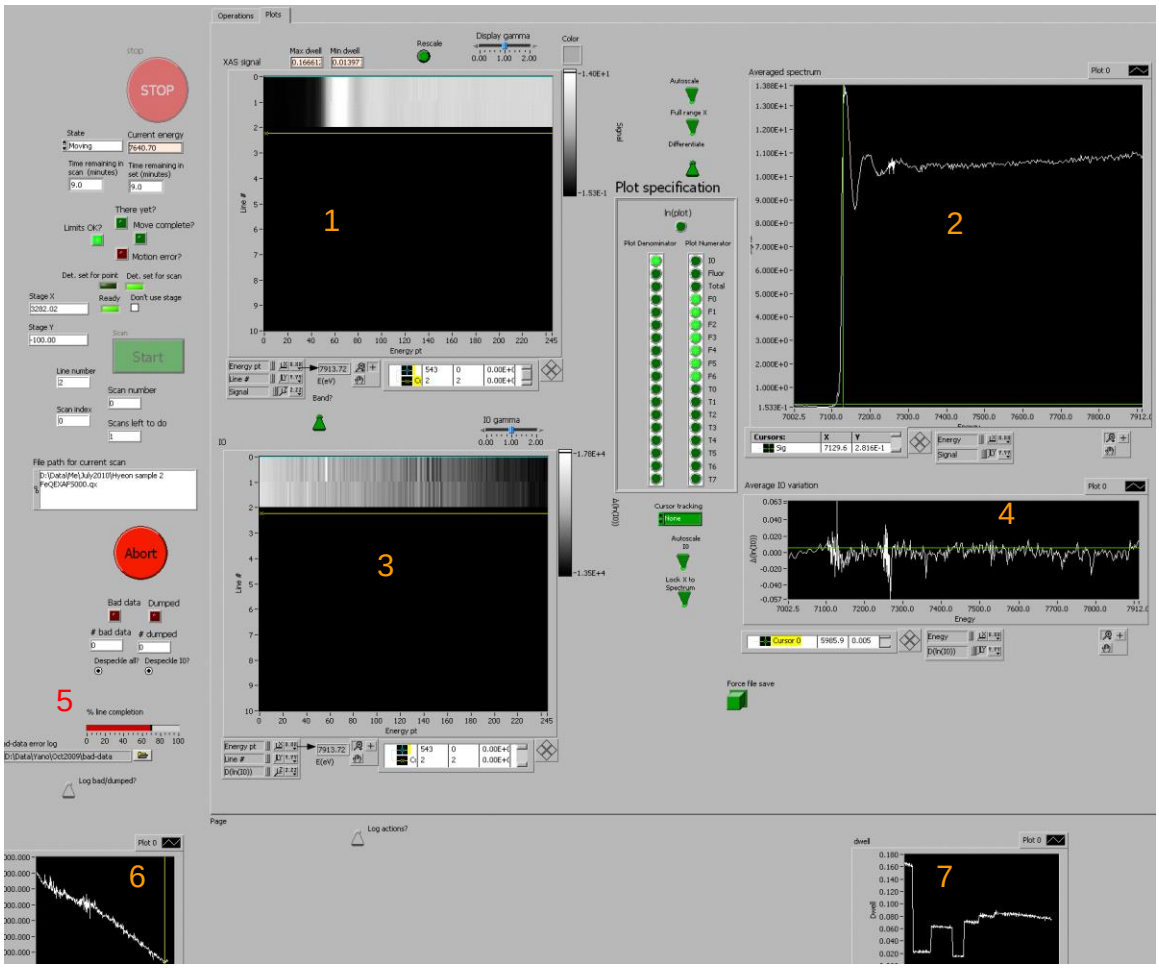

Figure 4. The Plots *page during data taking*

Data taking is started and stopped using the Start and Abort buttons as expected. The lines of data that have been taken so far in the current scan are shown as an intensity map labeled **1**. The horizontal axis is energy, or more accurately, energy point. Since the data are generally not uniformly tabulated in energy, each pixel in the horizontal represents a variable energy interval. The average of the lines between the blue and yellow cursors is shown in a conventional signal-vs.-energy plot in **2**. The same plotting controls are available here as in SXAS. Similarly, I_0 is shown in a 2D plot in **3**, with blue and yellow cursors that track those in the signal plot **1**. The reason for confining the average to the range between the cursors is that you may want to look for trends in the data, such as the signal level dropping because the sample is moving or the shape of the XANES changing due to radiation damage. The Band? switch lets you constrain the

spacing between the cursors to a constant (a control for the width becomes visible) so that you can compare, for instance the average of lines 0-2 and 10-12 just by moving one cursor. The Rescale button scales the display to fit all lines specified in the scan-def.

Plot **4** is an I_0-glitch plot just as in SXAS, but it operates on the average specified by the cursors in **1**. The I_0 for the line just completed is shown in **6**.

The % line completion indicator **5** shows where the energy is on a scale such that 0 corresponds to the beginning of the data range and 100 to the end. When the scan starts, this indicator is under-range because the scan includes a ramp-up interval which is not part of the acquired data.

The dwell time per point is shown in **7**. It is instructive to see how short the acquisition time is per point for each line. Dwell times less than ~20ms can result in lots of Bad Data events. These are like the ones you get in mapping and cause the line to have to be repeated. It appears that there is a bug somewhere in the XIA firmware or drivers which causes this problem, and which *may* get fixed when we go to the new XIA hardware.

There is a Force File Save button which will cause the program to save the data taken so far in the current scan. Unlike SXAS, QXAS only writes the data file when the scan is done, rather than writing the header before the data are taken. For debugging purposes, there are switches to set up logging of Bad Data events or beam dumps, and for logging program actions such as changing states. Users should not need these functions. The Despeckle? radio buttons implement an algorithm to correct (roughly) a known problem with the XIA hardware, the same problem which causes speckles to appear in maps of uniform areas. Leave these checked.

When boustrophedon mode is enabled, a control appears which specifies an energy shift to be applied to alternate lines to compensate for monochromator backlash.

IV. Data analysis considerations

The data files have a `.qx` extension and are to be read using the QXAS Editor, for which there is a separate manual. Deadtime correction parameters are defined in the scaler map and written into the data file, so there's no need to read in another file. It is tedious but important to inspect every fluorescence channel for glitches; again a reflection of intermittent bugs in the XIA hardware. These show up as white or black

lines in the 2D display and can be removed with the deglitching functions. If you want to average QXAS files with SXAS files, you have to use the Change Gain function in EXAFS Editor to adjust the I_0 scale so the signal matches. You also need to do Sum Scaler into Scaler on either the QXAS or SXAS files because the total fluorescence counts come in on scaler 2 for SXAS and scaler 1 for QXAS, and you need to have the fluorescence counts appear in a common scaler for both types in order for the averaging to work. When you write a `.dat` file out of QXAS Editor, you need to make sure that the blue and yellow cursors in the 2D display cover the whole data range. Noise analysis and measurements of time and total counts in EXAFS Editor will work on `.dat` files made in QXAS Editor.